IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Attorney Docket No. : 22080.002

In re Application of

Luis Alfredo Diaz Chavez Examiner : **Rebecca Y. Lee**

Serial No.: **10/565,715** Art Unit : **1793**

Filing Date **06/12/2006** Conf. No.: **7944**

For **CALCO-MAGNESIAN AQUEOUS SUSPENSION AND METHOD FOR THE PRODUCTION THEREOF**

DECLARATION

Mail Stop: Fee Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Madam:

My name is Marc PELLETIER, I am an engineering graduate of "Polytech'Orleans", section "Minerals and ores processing", since 1985, and Head of the Analytical Laboratory and Technical Assistance of Lhoist R&D Center, since 2001.

I have read the Specification in the above pending application and I am familiar with the claims in the case. I am also familiar with the *Langelin et al.* reference being cited by the Examiner (DE 4302539).

I performed a series of comparative laboratory tests measuring several parameters of samples made according to the teachings of the *Langelin et al.* reference.

The passage of page 3, l.. 7 to 11, of the Office Action suggests that "*it would have been obvious to one of ordinary skilled in the art at the time of the invention to have optimized the particle size and the specific surface area of Langelin, including the claimed particle size of less than or equal to 20 or 5 µm and the claimed specific surface area of less than or equal to 5 m²/g in order to achieve the desired viscosity*". Consequently, only factual evidence to show that optimizing the surface area or particle size in Langelin would lead a viscosity beyond the claim range or that the claimed surface area or viscosity would have in fact resulted in unexpected properties of the suspension could be convincing.

Therefore I have prepared milks of lime according to a method very similar to example 2 of Langelin. This is to say starting from QUICKLIME added to large excess of slaking water in such a way that a slurry is obtained ("wet" slaking) and is kept at a temperature around 40°C during its preparation (quicklime slaking). The detailed operating procedure for this milk of lime number 1 preparation according to example 2 of Langelin is as follows:
In a 2 dm³ beaker, I added 976 g of demineralised water, 12 g of a 50% sodium-polyacrylate solution, 12g of CaSO4.2H2O (eq. of 10g CaSO4.0,5H2O). This suspension was placed in a thermostatic bath at 20°C, then heated by inserting a thermal resistance until the suspension reached a temperature of 40°C. Quicklime was then added in order to maintain the temperature between 38 and 42°C. The system was mixed by hand, using a plastic rod. Quicklime was added until a concentration of about 60% was reached.

The viscosity of the slurry obtained as well as the reactivity (t90) of the suspension were then measured respectively according to the example 1 and the example 7 of the current invention. Then the particles size distribution by laser diffraction as well as BET specific surface area were measured on the solid particles of the slurry.

Milk of lime number 2 was obtained from milk of lime number 1, by wet milling of the solid particles of the slurry in order to approach as close as possible the particle

size according to the Applicant's invention. Milk of lime number 3 was prepared according to the operating procedure of milk of lime number 1 but by adding during the lime slaking a surface inhibitor known by the applicant in order to approach the specific surface area calculated according to the BET method and claimed in the Applicant's invention. Milk of lime number 4 was obtained by wet milling of milk of lime number 3 in order to approach the particle size distribution of the Applicant's invention. The detailed operating procedure for milk of lime 2 to 4 is given below.

Milk of lime number 2 :The milk of lime number 1 was milled in a small-size ball mill (Retsch PM400 - serial number 20.532.0001/8 1301 041) for 4 hours;

Milk of lime number 3 : In a 2 dm^3 beaker, I added 963 g of demineralised water, 12 g of a 50% sodium-polyacrylate solution, 12 g of CaSO4.2H2O (eq. of 10 g CaSO4.0,5H2O) and 13 g of CaCl2.2H2O (eq. of 10 g CaCl2). This suspension was placed in a thermostatic bath at 20°C, then heated by inserting a thermal resistance until the suspension reached a temperature of 40°C. Same quicklime as for milk of lime number 1 was then added in order to maintain the temperature between 38 and 42°C. The system was mixed by hand, using a plastic rod. Quicklime was added until a concentration of about 60% was reached.

Milk of lime number 4 :The milk of lime number 3 was milled in a small-size ball mill (Retsch PM400 - serial number 20.532.0001/8 1301 041) for 4 hours.

The results of the different measures on the four milks of lime and corresponding solid phases are given in the table 1 below:

		Langelin example 2	Langelin + wet milling	Langelin + surface inhibitor	Langelin + surface inhibitor + wet milling
Milk of lime number		#1	#2	#3	#4
d10	µm	3.5	1.0	7.5	1.0
d50	µm	250	3.0	324	4.5
d90	µm	796	8	>1000	30
d98	µm	>1000	20	>1000	46
BET	m^2/g	14	20	9	13
Concentration in solid	%	62	63	62	63
t90	s	78	8	94	34
Viscosity	cP	800	8495	1125	8455

Comments of table 1 :

1) Milk of lime #1. As a desired effect of the prior art of Langelin, the particle size distribution is very coarse with d98 higher than 1,000 µm and even a d50 as big as 250 µm (this is to say that half the solid particles has a size bigger than 250 µm). This particle size is a lot coarser than the one claimed for the present invention. The BET specific surface area of the solid phase of the slurry is 14 m^2/g, which is higher than the range claimed by the Applicant's invention. The viscosity of milk of lime # 1 is 800 cP, i.e. 0.8 Pa.s, for a solid mater content of 62 %. The reactivity of the milk of lime is by far too low. Indeed the time to reach 90% of the final conductivity (t90) according to the test described in example 7 of the present invention is of nearly 80 s, which is a too high duration compared with the about 2 s recited in table 7 of the present invention.

2) Milk of lime #2. In a first attempt to try to optimize the BET specific surface area and particle size distribution from Langelin as suggested in the Office Action a wet milling has been performed on milk of lime # 1 in order to obtain milk of lime # 2. Now the particle size distribution is greatly reduced (d98 of 20 µm instead of more than 1000 µm and a d50 divided by 100!) and is quite consistent with the present invention. But compared to the present invention, the BET specific surface area is still out of range and in a higher extent than milk of lime number 1! The reactivity of the milk of lime (8 s) is closer to the current invention target but its viscosity (8.5 Pa.s) is 10 times higher the one of milk of lime number 1. This is to say far above the

1.2 Pa.s expected by the current invention. Such a milk of lime would no longer be handable and pumpable.

3) Milk of lime #3. In an attempt to reduce the BET specific surface area from Langelin teaching, a surface inhibitor known by the applicant was added during the lime slaking according to example 2 of Langelin . Now the BET specific surface area is of 9 m^2/g, so in the range claimed by the Applicant's invention. But the particle size distribution is coarser than milk of lime #1, with d98 and d90 higher than 1,000 μm and even a d50 as big as 324 μm (this is to say that half the solid particles has a size bigger than 324 μm). This particle size is again a lot coarser than the one claimed for the present invention. The viscosity of milk of lime # 3 is 1125 cP, i.e. 1.1 Pa.s for solid mater content of 62 %; this viscosity is still consistent with the current invention but close to the higher range value of 1.2 Pa.s. On the contrary, the reactivity of the milk of lime is by far too low and lower than milk of lime # 1. Indeed the time to reach 90% of the final conductivity according to the test described in example 7 of the current invention is of more than 90 s, which is a too high duration compared with the about 2 s recited in table 7 of the current invention.

4) Milk of lime #4. In a second attempt to try to optimize the BET specific surface area and particle size distribution from Langelin as suggested by the Office Action, a wet milling has been performed on milk of lime #3 to obtain milk of lime #4 . Now the particle size distribution is greatly reduced (d98=46 μm) but not as far as expected by the present invention. Compared to the present invention, the BET specific surface area is now out of range (13 m^2/g) even if it was not the case of the initial milk of lime number #3 (before milling). The reactivity of the milk of lime (34 s) is too high compared to the target expected by the present invention and its viscosity (8.5 Pa.s) is 10 times higher the one of milk of lime number 1. This is to say far above the 1.2 Pa.s expected by the current invention. Such a milk of lime would no longer be handable and pumpable.

One can see that there is no way to optimize the particle size distribution and BET specific area of Langelin to obtain Applicant's invention i.e with small particle size and low BET specific area at the same time in order to give a reactive milk of lime of low viscosity. By an attempt to improve (lower) the particle size distribution, the BET specific surface area got worse (higher). Conversely, by an attempt to improve (lower) the BET specific surface area, the particle size distribution got worse (bigger). From Langelin's teaching, no "optimisation" of BET specific area and particle size distribution is possible. Moreover, there is either a good viscosity and a poor reactivity or a good reactivity and a poor viscosity.

In a last attempt to reduce the BET specific surface area from Langelin teaching, the milk of lime #1 (example 2 of Langelin) was reproduced (now as milk of lime #5) and then the solid particles were sieved at 200 µm and only the retained particles were taken, as suggested in page 6, line 54 of DE 4302539 A1 of the German text of Langelin in order to produce milk of lime #6. The results are shown in table 2, wherein example 2 of Langelin has been reproduced (milk of lime #5). The results are shown next to the results of milk of lime #1 already communicated for comparison purpose. We can then see the repeatability of the tests. Concerning the particle size distribution, the one of milk of lime #5 is broader than for milk of lime #1, with lower average value (d50) and higher d90 (more coarser particles than in milk of lime #1). Globally, milks of lime #1 and #5 have solid particles of big size, a lot coarser than the range claimed in the Applicants' invention. Concerning the BET specific surface area, the two values are of 14m²/g. The reactivity test (t90) as well as the viscosity of milk of lime #5 show also very consistent results with milk of lime #1.

Milk of lime #6 comes from milk of lime #5 by wet sieving on 200 µm sieve and keeping the retained on the sieve. As expected by the reading of Langelin, a decrease of the BET specific surface area is recorded compared to solid particles of milk of lime #5 (10m²/g instead of 14). On the other hand, by keeping the retained on the 200 µm sieve, the particle size distribution of the solid increased significantly, the d50

being now at 500 µm. Unfortunately, it was not possible to further make a milk of lime on the basis of this> 200 µm solid. An attempt to make a milk of lime of about 60% of solid matter (SM) like the original milk of lime #5, leads to an absorption of the whole quantity of water by the solids so that the sample looks a "wet solid", (see picture 1 below), without "free" water. A further attempt to dilute more in order to obtain 40% solid suspension, leads to two separate phases product. The solid remains as such at the bottom with the liquid water over ; the situation is such as if we want to add water on a certain quantity of wet sand (see picture 2). As a conclusion, it is really impossible to make a true slurry with the over 200 µm solids particles. This last trial shows that an attempt to decrease specific surface area makes the solid particle coarser so more far from the current invention with in addition, the impossibility to further use this solid to prepare a milk of lime.

		Langelin example 2	Langelin example 2bis	Langelin example 2bis > 200µm
Milk of lime number		#1	#5	#6
d10	µm	3.5	2.2	
d50	µm	250	93	500
d90	µm	796	946	
d98	µm	>1000	>1000	
BET	m^2/g	14	14	10
Concentration in solid	%	62	64	-
t90	s	78	76	-
Viscosity	cP	800	880	-

This last experimentation shows that, in order to obtain the expected BET specific surface area according to the present invention the granulometric sizes of the particles of the Langelin slurry must be selected in a preferable range higher than 200 µm. However after this selection there is no more slurry at all. If the totality of the particles is maintained, the BET surface area becomes immediately higher than 10 m^2/g.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

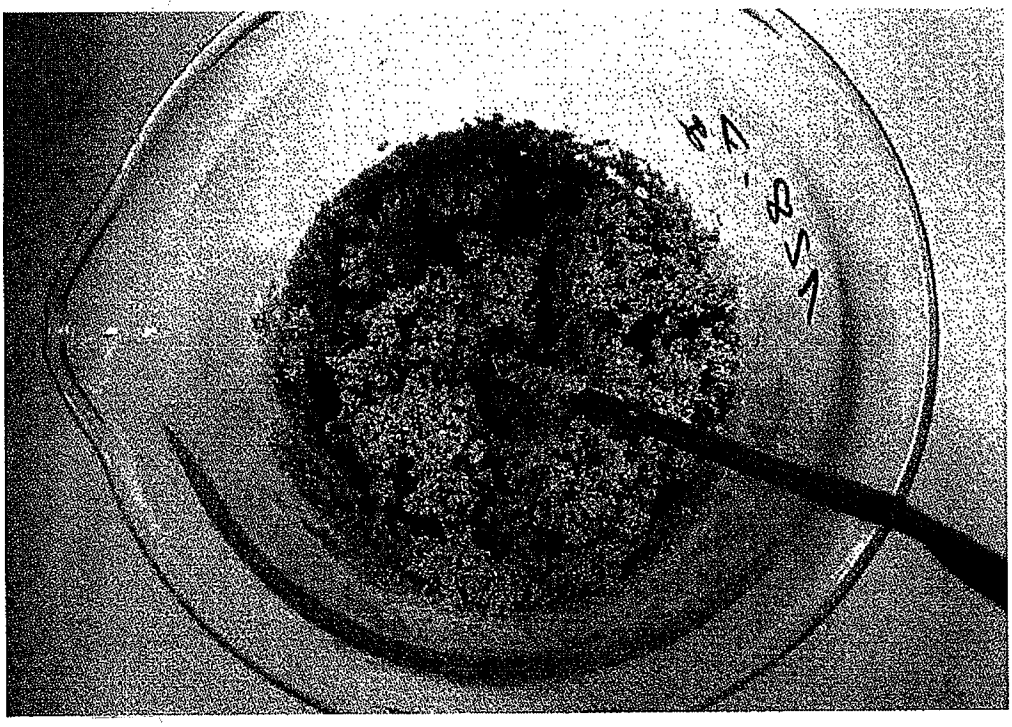
Marc PELLETIER

November 9, 2011

Signature

Marc PELLETIER

Picture 2



Picture 1